GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. ____

Project No. A-3454 GTRI/~~GIT~~ DATE 1/13/83

Project Director: Joe N. Harris ~~School~~/Lab EMSL

Sponsor: Selenia S.p.A (Rome, Italy) Co

Type Agreement: Std. Research Project Agreement & P.O. #018209882

Award Period: From 1/1/83 To 12/31/83 (Performance) 12/31/83 (Reports)

Sponsor Amount: Total Estimated: $ 14,843 Funded: $ 4,788

Cost Sharing Amount: $ NA Cost Sharing No: ____

Title: Characterization of Properties of High Purity Fused Silica Slips

ADMINISTRATIVE DATA

OCA Contact Frank H. Huff

1) Sponsor Technical Contact:

Mr. Robero Brugiotti
Selenia S.p.A
Via Tirburtina
KM 12.400
00131 Rome, Italy

Telefono 43601 Telex 613690

2) Sponsor Admin/Contractual Matters:

Mrs. Ardia, Piva
Selenia S.p.A.
Via Tirburtina
KM 12.400
00131 Rome, Italy

Defense Priority Rating: ____

Military Security Classification: ____

(or) Company/Industrial Proprietary: ____

RESTRICTIONS

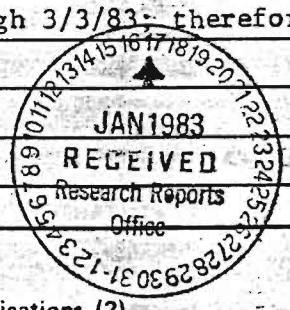
See Attached ____ Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval – Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with No

COMMENTS:

Project is funded for one (1) characterization only. A letter report is due by March 2, 1983. Selenia has provided a Letter of Credit valid only thorugh 3/3/83; therefore first report must be submitted by that time.



COPIES TO:

Research Administrative Network
Research Property Management
Accounting
Procurement/EES Supply Services
~~Research Security Services~~
Reports Coordinator (OCA)
GTRI
Library
Research Communications (2)
Project File
Other Greg Dew
Other ____

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date February 20, 1984

Project No. A-3454 ~~School~~ Lab EMSL

Includes Subproject No.(s)

Project Director(s) Joe N. Harris GTRI / ~~GIT~~

Sponsor Selenia S.p.A. (Rome, Italy)

Title Characterization of Properties of High Purity Fused Silica Slips

Effective Completion Date: 12/31/83 (Performance) 12/31/83 (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [x] Final Invoice or Final Fiscal Report
- [ ] Closing Documents
- [ ] Final Report of Inventions
- [ ] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other

Continues Project No. Continued by Project No.

COPIES TO:

Project Director
Research Administrative Network
Research Property Management
Accounting
Procurement/EES Supply Services
~~Research Security Services~~
Reports Coordinator (OCA)
~~Legal Services~~

Library
GTRI
Research Communications (2)
Project File
Other

Form OCA 60:1028

A-3454

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

February 22, 1983

Selenia Industrie Electtroniche Associate SpA
Via Tirbutina
Km. 12.400
P. O. Box 7083
00100 Rome, Italy

Attention: Mr. Cesarotti, Technical Manager

Subject: Technical Letter Report: Characterization of High Purity Fused Silica Slip 053182-B-HP

Gentlemen:

The rheological characteristics of the high purity fused silica slip 053182-B-HP from Thermo Materials Corporation, Atlanta, Georgia, were evaluated to determine the acceptability of the slip per specifications of Selenia Industrie Elettroniche Associate SpA.

The high purity slip was packaged in a five gallon polyethylene carboy with a diameter of 38.9 centimeters. The speed of the roll mill was adjusted to produce a surface velocity of the carboy of one meter per second. After a rolling time period of ninety-six hours, measurements were made for solids content, viscosity and pH.

Thirty, 19.1 millimeter diameter test bars were cast in new plaster molds. The test bars were dried for twenty-four hours at 150° C. Test bar sets of ten (10) each were sintered at temperatures of 1210° and 1221° C for four hours. The sintered bars were used to determine dynamic elastic modulus, modulus of rupture, bulk density, porosity and cristobalite content. The slip test procedures were as follows:

I. Solids Content. The solids content was measured by evaporating the liquid from approximately 50-grams of slip to a constant weight in a 150° C oven.

II. pH. The pH was measured by immersing the glass electrodes of the pH meter directly in the slip which had been temperature stabilized to 22° C.

III. Viscosity. The apparent viscosity was measured with a model LVF Brookfield Viscometer with a number 2 spindle.

Measurements were made at speeds of 6, 12, 30 and 60 rpm. The temperature of the slip was stabilized at 22° C.

IV. Particle Size Distribution. The particle size distribution was measured by Micromeritics Instrument Corporation (MIC) Atlanta, Georgia using the MIC Sedigraph 5000 and was supplied by the vendor. Data are provided as a plot of cumulative mass percent versus equivalent spherical diameter.

V. Chemical Composition. These data were determined by Spectro Chemical Laboratories, Pittsburgh, Pennsylvania, and were provided by the vendor.

VI. Bulk Density and Porosity. These properties were determined in accordance with ASTM C373 "Standard Method of Test for Water Absorption, Bulk Density, Apparent Porosity and Apparent Specific Gravity."

VII. Elastic Modulus. This property was determined in accordance with ASTM C623 "Standard Method of Test for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass Ceramics by Resonance."

VIII. Modulus of Rupture. This property was determined by a procedural modification of ASTM C158 "Flexure Testing of Glass." The test bars were broken in four point, 1/4-point configuration with a 102 millimeter lower span and a 51-millimeter upper span. The loading rate of the hydraulic Universal Test Machine was 600 pounds (272.4 kilograms) per minute.

IX. Cristobalite Content. The percentage of α cristobalite contained in the sintered specimens was determined by an x-ray analysis technique developed by the Georgia Institute of Technology. The results are based on a comparison with an industry accepted standard, Georgia Tech A-4.

The property measurements made on the slip are shown in Table I. The particle size distribution from Micromeritics Instrument Company and the chemical analysis from Spectrochemical Laboratories are reproduced from the original copies supplied as attachments I and II.

TABLE I
SLIP PROPERTIES FOR THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP 053182-B-HP

Solid Content (%)	82.8
pH @ 22° C	4.7
Viscosity @ 22° C (centipoise)	
@ 6 rpm	210
12 rpm	170
30 rpm	143
60 rpm	130
Mean Particle Size (μm)	7.8

The sintering operations were performed in a bottom-loading electric-resistance furnace with the bars in a vertical position supported in a fused silica foam block. The sintering schedule was programmed by microprocessor control and is shown in Table II.

The test results for the physical and mechanical properties obtained from the two sintering temperatures, 1210° and 1221° C, are shown in Table III.

The qualification test results for the two firings provided to Mr. Righetti are appended as Attachments III and IV. This slip is satisfactory for radome manufacture.

TABLE II

SINTERING SCHEDULE FOR HIGH PURITY
SLIP-CAST FUSED SILICA TEST BARS

Room Temperature to 927° C	10 hours
Hold at 927° C	6 hours
927° C to 1038° C	90 minutes
Hold at 1038° C	2 hours
1038° C to Sintering Temperatures	90 minutes
Hold at Sintering Temperature	4 hours
Turn off Kiln and Cool Bars in Kiln	

*Typical, can vary.

**Sintering #1 - 1221 C
Sintering #2 - 1210 C

TABLE III

PHYSICAL AND MECHANICAL PROPERTIES OF
TEST SPECIMENS SINTERED 4 HOURS

	Sintering Temperature	
	1221° C	1210° C
Elastic Modulus (10^6 psi)	5.65 ± 0.13	5.65 ± 0.06
Modulus of Rupture (10^6 psi)	6.50 ± 0.67	5.89 ± 0.48
Bulk Density (g/cm^3)	1.959 ± 0.003	1.951 ± 0.002
Porosity (%)	10.61 ± 0.11	10.89 ± 0.06
Cristobalite Content (%)		
Strongest Bar	0.6	0.5
Weakest Bar	1.3	0.6

Respectfully submitted,

Joe N. Harris
Project Director
Senior Research Engineer

jw

PARTICLE SIZE DISTRIBUTION

SAMPLE IDENTIFICATION Thermo Materials Mal # 999

DATE 6-10-82

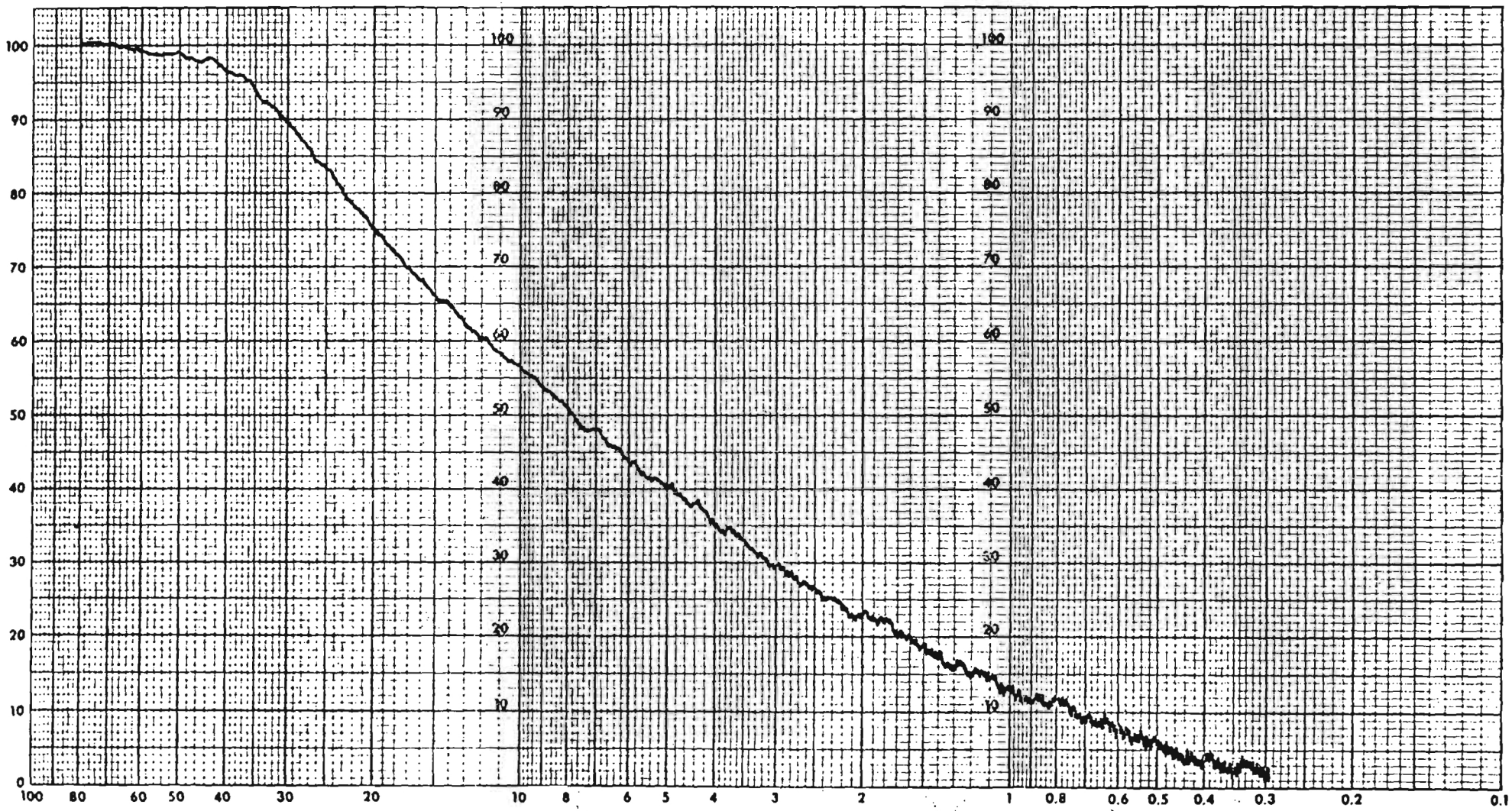
Density 2.2 g/cc LIQUID Calgon water Density 0.9954 g/cc Viscosity 0.7840 cp

BY SD

Preparation Silica Slurry 053182 B-HP1

TEMPERATURE 31 °C

RATE 833 START DIA. 80 µm



ATTACHMENT I.

micromeritics® instrument corporation

ATTACHMENT II.

Spectrochemical Laboratories Inc.

Telephone: 412-371-2345
TWX: 710-664-4439

8350 FRANKSTOWN AVENUE
PITTSBURGH, PA. 15221

June 9, 1982
Sample Recd. 6-3-82

Thermo Materials Corporation
3449 Church Street - P.O. Box 925
Scottdale, Georgia 30079

Attn: Mr. Donald Kenagy

ANALYSIS REPORT
IN WEIGHT %

OUR:W 7903

99.5% + SiO2
6-2-82

	-2 053182 B-HP
Fe2O3	.003
Al2O3	.23
CaO	.001
MgO	.007
TiO2	.002
Cr2O3	.001
CoO	*.001
SiO2	99.76

ANALYSIS REPORT
IN PPM

Na	*10
K	*10
Li	*10

*Not detected. The number indicates the minimum limit of detection.

SPECTROCHEMICAL LABORATORIES, INC.,

I.L. Fulton

ATTACHMENT III.

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

FUSED SILICA SLIP
QUALIFICATION TEST RESULTS

DATE: 15 Feb 83

SLIP NO. 053182-B-HP

1. PH 4.7
2. VISCOSITY 143 @ 30 RPM, SPINDLE NO. 2, 22 ° C.
3. SOLID CONTENT 82.8 %

PROPERTIES FOR SINTERED TEST BARS @ DENSITY 1.94-1.95 g/cm^3

#1-#10 NUMBER OF BARS 10 FIRING TIME 4 hours TEMP 1221 ° C.

1. DENSITY 1.959 ± 0.003 g/cm^3
2. MOR 6495 ± 673 psi
3. MODULUS OF ELASTICITY $5.654 \pm 0.133 \times 10^6$ psi (2 bars)
4. CRISTOBALITE CONTENT 0.6 and 0.5 %
5. POROSITY 10.61 ± 0.11 %

COMMENTS AND RECOMMENDATIONS Based on available data this slip is satisfactory for the manufacture of SCFS radomes

RESULTS VERIFIED BY: _____

DATE: 15 Feb 83

NOTEBOOK: PAGES 118-129

INCLUSIVE DATES 12/7/82 - 2/15/83

ATTACHMENTS: CHEMICAL ANALYSIS BY SPECTROCHEMICAL LABORATORIES FOR TMC
PARTICLE SIZE DISTRIBUTION BY MICROMERITCS INSTRUMENT CO FOR TMC

ATTACHMENT IV.

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

FUSED SILICA SLIP
QUALIFICATION TEST RESULTS

DATE: 15 Feb 83

SLIP NO. 053182-B-H8

1. PH 4.7
2. VISCOSITY 143 @ 30 RPM, SPINDLE NO. 2, 22 ° C.
3. SOLID CONTENT 82.8 %

PROPERTIES FOR SINTERED TEST BARS @ DENSITY 1.94-1.95 g/cm^3

#11-#20 NUMBER OF BARS 10 FIRING TIME 4 hours TEMP 1210 ° C.

1. DENSITY 1.951 ± 0.002 g/cm^3
2. MOR 5893 ± 482 psi
3. MODULUS OF ELASTICITY $5.646 \pm 0.057 \times 10^6$ psi
4. CRISTOBALITE CONTENT 2 samples 1.3 and 0.6 %
5. POROSITY 10.89 ± 0.06 %

COMMENTS AND RECOMMENDATIONS Based on available data this slip is satisfactory for the manufacture of SCFS radomes

RESULTS VERIFIED BY: _____

DATE: 15 Feb 1983

NOTEBOOK: PAGES 118-129

INCLUSIVE DATES 12/7/82 - 2/8/83

ATTACHMENTS: CHEMICAL ANALYSIS BY SPECTROCHEMICAL LABORATORIES FOR TMC
PARTICLE SIZE DISTRIBUTION BY MICROMERITICS INSTRUMENT CO FOR TMC

A-345

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

October 5, 1983

Selenia Industrie Electroniche Associate SpA
Via Tirbutina
Km. 12.400
P. O. Box 7083
00100 Rome, Italy

Attention: Mr. Cesarotti, Technical Manager

Subject: Technical Letter Report: Characterization of High Purity Fused Silica Slip 053182-B-HP

Gentlemen:

The rheological characteristics of the high purity fused silica slip 090183A from Thermo Materials Corporation, Atlanta, Georgia, were evaluated to determine the acceptability of the slip per specifications of Selenia Industrie Elettroniche Associate SpA.

The high purity slip was packaged in a five gallon polyethylene carboy with a diameter of 38.9 centimeters. Since this batch of slip was blended from several mills at Thermo Materials over a period of time, the normal fast rolling pre-treatment before use was not necessary. The slip was rolled in the carboy at 5 rpm for 24 hours before any measurements were made.

Thirty, 19.1 millimeter diameter test bars were cast in new plaster molds. The test bars were dried for twenty-four hours at 150° C. Three Test bar sets of ten (10) each were sintered at temperatures of 1205°, 1210° and 1213° C for four hours. The sintered bars were used to determine dynamic elastic modulus, modulus of rupture, bulk density, porosity and cristobalite content. The slip test procedures were as follows:

I. Solids Content. The solids content was measured by evaporating the liquid from approximately 50-grams of slip to a constant weight in a 150° C oven.

II. pH. The pH was measured by immersing the glass electrodes of the pH meter directly in the slip which had been temperature stabilized to 22° C.

III. Viscosity. The apparent viscosity was measured with a model LVF Brookfield Viscometer with a number 2 spindle. Measurements were made at speeds of 6, 12, 30 and 60 rpm. The temperature of the slip was stabilized at 20° C.

IV. Particle Size Distribution. The particle size distribution was measured by Micromeritics Instrument Corporation (MIC) Atlanta, Georgia using the MIC Sedigraph 5000 and was supplied by the vendor. Data are provided as a plot of cumulative mass percent versus equivalent spherical diameter.

V. Chemical Composition. These data were determined by Spectro Chemical Laboratories, Pittsburgh, Pennsylvania, and were provided by the vendor.

VI. Bulk Density and Porosity. These properties were determined in accordance with ASTM C373 "Standard Method of Test for Water Absorption, Bulk Density, Apparent Porosity and Apparent Specific Gravity."

VII. Elastic Modulus. This property was determined in accordance with ASTM C623 "Standard Method of Test for Young's Modulus, Shear Modulus, and Poisson's Ratio for Glass and Glass Ceramics by Resonance."

VIII. Modulus of Rupture. This property was determined by a procedural modification of ASTM C158 "Flexure Testing of Glass." The test bars were broken in four point, 1/4-point configuration with a 102 millimeter lower span and a 51-millimeter upper scan. The loading rate of the hydraulic Universal Test Machine was 600 pounds (272.4 kilograms) per minute.

IX. Cristobalite Content. The percentage of α cristobalite contained in the sintered specimens was determined by an x-ray analysis technique developed by the Georgia Institute of Technology. The results are based on a comparison with an industry accepted standard, Georgia Tech A-4.

The property measurements made on the slip are shown in Table I. The particle size distribution from Micromeritics Instrument Company and the chemical analysis from Spectrochemical Laboratories are reproduced from the original copies supplied as attachments I and II.

The sintering operations were performed in a bottom-loading electric-resistance furnace with the bars in a vertical position supported in a fused silica foam block. The sintering schedule was programmed by microprocessor control and is shown in Table II.

The test results for the physical and mechanical properties obtained from the three sintering temperatures, 1205°, 1210° and 1213° C, are shown in Table III.

TABLE I

SLIP PROPERTIES FOR THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP 053182-B-HP

Solid Content (%)	82.8
pH @ 20° C	5.3
Viscosity @ 20° C (centipoise)	
@ 6 rpm	220
12 rpm	187
30 rpm	159
60 rpm	153
Mean Particle Size (μm)	8.0

TABLE II

SINTERING SCHEDULE FOR HIGH PURITY
SLIP-CAST FUSED SILICA TEST BARS

Room Temperature to 927° C*	10 hours
Hold at 927° C*	6 hours
927° C to 1038° C	90 minutes
Hold at 1038° C	2 hours
1038° C to Sintering Temperatures**	90 minutes
Hold at Sintering Temperature	4 hours
Turn off Kiln and Cool Bars in Kiln	

*Typical, can vary.

**Sintering #1 - 1210° C
Sintering #2 - 1205°/C
Sintering #3 - 1213°/C

TABLE III

PHYSICAL AND MECHANICAL PROPERTIES OF
TEST SPECIMENS SINTERED 4 HOURS

	Sintering Temperature		
	1205° C	1210° C	1213° C
Elastic Modulus (10^6 psi)	5.45 ± 0.08	5.66 ± 0.07	5.78 ± 0.08
Modulus of Rupture (10^3 psi)	6.50 ± 0.67	5.08 ± 0.99	4.17 ± 0.11
Bulk Density (g/cm^3)	1.938 ± 0.001	1.945 ± 0.002	1.945 ± 0.003
Porosity (%)	10.96 ± 0.09	10.70 ± 0.08	10.9 ± 0.34
Cristobalite Content (%)			
Strongest Bar	0.7	0.5	0.7
Weakest Bar	1.3	0.7	1.0

DISCUSSION:

This slip and the test bars made therefrom meet the technical specifications as set down by Selenia. The pH of 5.3 falls in the acceptable range of 3.5 to 5.5. The 30 rpm Brookfield viscosity of 159 centipoise is less than the maximum allowable 225 centipoise. The solids content of 82.8 percent falls within the allowable range of 82 to 83 percent. The SiO_2 content of 99.77 percent exceeds the required 99.5 percent SiO_2. The maximum total alkali metals (Na, K, Li) of 36 ppm is less than the allowable 50 ppm. The alkaline earths (CaO and MgO) total 140 ppm and are well under the allowable 300 ppm. The mean particle diameter of 8.0 or 8.1 µm (the curve is difficult to read exactly because of a perturbation at the 50 cumulative mass percent level) falls in the acceptable range of 7-8.5 µm. The percentage of particles with diameters less than 2 µm was 24, which is less than the permitted maximum of 30 percent.

Four hour firings at 1205, 1210 and 1213° C brought all three sets of test bars into the specified 1.94 to 1.95 gm/cm^3 bulk density range and all three sets of test bars exceeded the five million psi requirement for modulus of elasticity and 4000 psi or greater for modulus of rupture. The cristobalite content on strongest and weakest test bars from each group was less than the maximum permitted, two percent.

Based on the above Selenia specifications this slip meets all the requirements for radome manufacture. However, this slip had some peculiarities not experienced with recent Thermo Mateials high purity fused silica slip. The pH, although within the acceptable range based on Selenia's specifications was considerably higher than recently characterized slips. Most recently characterized slips have had pH values in the range of 4.5 to 4.8. This might be an indication of a lack of fine particles in this slip, however, the slip sintered to the correct density at a rather low firing temperature (1205 to 1213° C). These sintering conditions point to a material with a large surface area, hence an adequate amount of fine material. Also the mean modulus of rupture for the first two set of bars was above the average with respect to density for slips characterized for Selenia in the past seven years.

The major point of concern with the modulus of rupture data was the large 95 percent confidence intervals on all three sets of bars. These values were 718, 988 and 1053. The previous high 95 percent confidence interval for any set of ten bars was 673. The average mean value for 95 percent confidence interval for previous ten bar sets was 357. The reason for the high scatter in MOR data is not known at this time.

The percent of cristobalite measured in the current test bars is higher with resect to bulk density than for any other previous slip. This higher percentage of cristobalite may be partly responsible for the large data scatter.

RECOMMENDATIONS:

Since this slip batch (090183A) meets all of the current specifications for high purity fused silica slip, it must be accepted as material suitable for fabricating slip-cast fused silica radomes. However, if problems develop with the fired properties of hardware prepared from this slip then it is suggested that the specifications for fused silica slip and hardware prepared therefrom be amended prior to purchasing any additional materials.

The following suggested specification changes should be considered:

1. Since this is the only batch of slip examined in the past seven years to indicate a detectable quantity of sodium and potassium, considerations should be given to tightening the alkali metals total to 30 ppm. That is the same as saying sodium, potassium and titanium must not be detected.

2. Based on data obtained over the past several years, the specification for modulus of rupture should be changed to require ten test specimens, 3/4 inch in diameter, broken in 4-point, 1/4 point loading on a four inch span. For bars with a bulk density of 1.94-1.95 g/cm^3 minimum acceptable MOR should be 4300 psi with a 95 percent confidence interval not to exceed plus or minus 600 psi.

3. The maximum acceptable cristobalite content for test bars of less than 1.95 g/cm^3 density should be reduced to one percent.

Respectfully submitted.

Joe N. Harris
Project Director

jw